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Evaluation of Technologies for Treating Aqueous Metal/Cyanide Bearing Hazardous Waste (F007)

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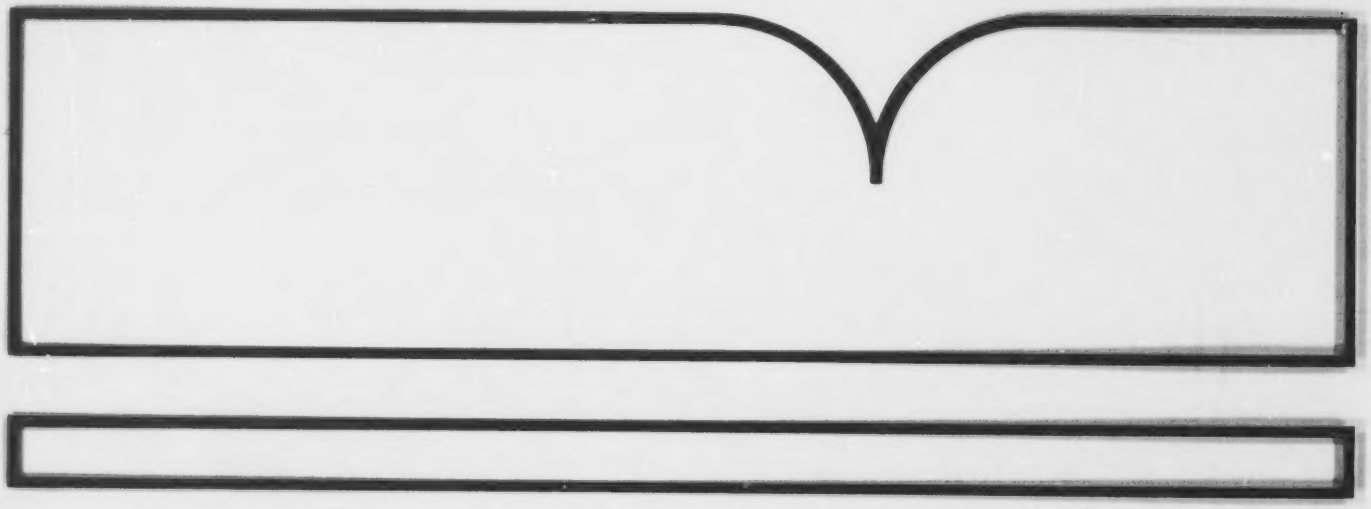

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EVALUATION OF TECHNOLOGIES FOR TREATING AQUEOUS METAL/CYANIDE BEARING HAZARDOUS WASTE (F007)

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EPA Contract 68-03-4038

EPA Project Officer
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16. ABSTRACT

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ABSTRACT

As a result of recent developments in the area of hazardous waste management, the U.S. Environmental Protection Agency is evaluating the performance of various technologies for the treatment and/or destruction of certain wastes that are presently being disposed of in landfills and surface impoundments. As a part of this program the University of Cincinnati, at the US EPA Test and Evaluation Facility in Cincinnati, Ohio, is testing existing treatment technologies that are applicable to metal- and cyanide-bearing hazardous wastes. The following unit processes have been evaluated: alkaline chlorination, precipitation, filtration, anion exchange and cation exchange. Results of these studies are presented.

INTRODUCTION

In support of the continuing need for research demonstrating and evaluating alternative technologies for the treatment of hazardous wastes, the USEPA's Test & Evaluation Facility, in Cincinnati, Ohio, has been selected as a center for the conduct of hazardous waste treatment technology assessment studies. The Hazardous Waste Engineering Research Laboratory (HWERL) in conjunction with the University of Cincinnati (UC), the on-site contractor, is providing technical support to carry out the fabrication, installation, and assessment of conventional unit processes. One major subject of interest concerns the treatment of spent process wastewater resulting from cyanide electroplating operations. Aqueous solutions with free and complexed metal cyanides are of major environmental concern as long as spent cyanide process solutions are generated. It becomes important to evaluate the various treatment options available. Unit processes typically used for the treatment of these metal cyanide wastes are: alkaline chlorination, electrolytic oxidation, ozonation, and to a lesser extent, wet air oxidation and ion exchange. Typically, cyanide destruction is followed by metals precipitation and effluent polishing. The work to be presented in this paper will concentrate on alkaline chlorination as the major technology around which modifications are made to obtain desirable treatment of the waste.

Alkaline chlorination of dilute (<1000 ppm) metal cyanide waste streams is a treatment technology which has been employed over twenty-five years, and the process has proven to be successful in destroying free

cyanides in aqueous solutions. The mechanism responsible for the destruction of cyanide via alkaline chlorination is illustrated in the following way:

(1) $CN^- + H_2O + OCl^- \rightarrow Cl\text{-}CN + 2OH^-$

where CN^- is converted to dissolved cyanogen chloride in the presence of hypochlorite.

(2) $Cl\text{-}CN + 2OH^- \rightarrow OCN^- + Cl^- + H_2O$

hydrolysis of cyanogen chloride

(3) $2OCN^- + 3OCl^- + H_2O \rightarrow 2CO_2 + N_2 + 3Cl^- + 2OH^-$

conversion to carbon dioxide, nitrogen and chlorine.

Alkaline chlorination of metal cyanide wastes is typically performed in two stages. During the first stage solution, pH is initially raised to 10 or higher. Hydrolysis of the cyanogen chloride complex is very rapid. During the second stage, the pH of the solution is reduced to 8.5 to complete the oxidation of cyanide to carbon dioxide and nitrogen.

Complexed (nonamenable) cyanides however are often left untreated and pass into precipitation processes which generate metal hydroxide/cyanide sludges or are discharged as final effluents. It is preferable to destroy cyanide rather than to have it mixed with metal hydroxide sludges which, in most cases, are land disposed. Further treatment may be required to remove these undesirable residuals.

In this study, a conventional treatment train was modified by the addition of other applicable technologies to avoid the limitations of alkaline chlorination in treating complexed metal cyanides. These technologies were anion exchange and precipitation of the ferrocyanide complexes in the form of ferri-ferrocyanide (Prussian Blue). Metals from the waste stream were treated using lime precipitation and cation exchange. Rearrangements of the unit processes were also evaluated for the purpose of segregating metals and cyanide in the sludge. Results of bench-scale studies were used to design the pilot-scale system.

APPROACH

The objective of this study is to evaluate the most effective way to remove (destroy) hazardous components from metal cyanide waste streams using the process of alkaline chlorination and necessary polishing steps. A configuration (treatment train) of unit processes was evaluated based on the ability to optimize treatment. Performance was evaluated at prescribed design parameters (ORP, pH, dosage, retention times). The data generated by this project will benefit commercial manufacturing operations utilizing on-site waste treatment systems, off-site treatment storage, and disposal facilities and regulatory agencies (federal, state, and local) in understanding the parameters which influence alkaline chlorination.

Initially, bench scale studies were conducted for the purpose of selecting the most effective treatment train in meeting the following treatment objectives:

* highest quality final effluent devoid of metal and/or cyanide constituents
* least amount of cyanide present in metal hydroxide sludges

Then process optimization studies were conducted to optimize the pilot-scale process parameters. Finally, performance evaluation tests were conducted on the pilot-scale units of interest. Data are presented from both types of studies.

WASTE DESCRIPTION

An estimated 7.9 billion gallons of toxic metal-bearing waste and 4.7 billion gallons of cyanide wastes were generated in the United States in 1983 (Ref. 1). The majority of metal/cyanide wastes generated are characteristic toxic metal wastes (RCRA Code Dxxx) and wastes that result from metal treating processes (RCRA Code Fxxx). Emphasis has been placed on the "F" series of wastes which are identified as follows (Ref. 2):

F006: Wastewater treatment sludges from electroplating operations except from the following processes: 1) sulfuric acid anodizing of aluminum; 2) tin plating of carbon steel; 3) zinc plating (segregated basis) on carbon steel; 4) aluminum or zinc-aluminum plating on carbon steel; 5) cleaning/stripping associated with tin, zinc, and aluminum plating on carbon steel; and 6) chemical etching and milling of aluminum.

F007: Spent cyanide plating bath solutions from electroplating operations (except for precious metals electroplating spent cyanide plating bath solutions).

F008: Plating bath sludges from the bottom of plating baths from electroplating operations where cyanides are used in the process (except for precious metals electroplating plating bath sludges).

F009: Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used in the process (except for precious metals electroplating plating bath solutions).

Figure 1 shows a schematic of how each of these waste codes is generated from a typical electroplating process. The RCRA waste code which will be evaluated for treatment is F007.

Twelve 55-gal drums of F007 electroplating waste were acquired from a local electroplating job shop. Drag-out from plating operations was added to give specified concentrations of cyanide and heavy metals. Results from the characterization of this waste are provided in Table 1.

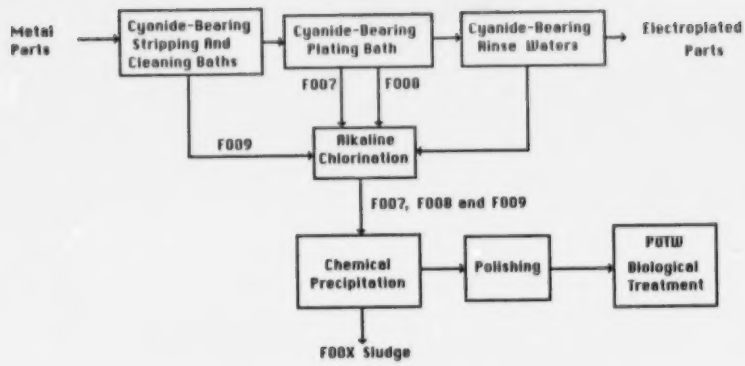

Figure 1. Typical Electroplating Process

TECHNOLOGY ASSESSMENT STUDIES

Assessment of the bench-scale and pilot-scale alkaline chlorination of a F007 electroplating waste is based upon: (1) a comprehensive waste characterization; (2) bench-scale process optimization studies and (3) monitoring of significant process parameters during pilot-scale operation. The raw waste was analyzed for cyanide, inorganics and organics in order to determine applicable treatment options. From these data, five treatment trains were identified as having potential for treatment of the characterized waste. These five options were evaluated during bench-scale process optimization studies. Not only were configurations of various unit processes optimized but, also chemical dosages, ORP, pH and hydraulic

Table 1

Characterization Data for the Raw Waste (F007)

Pollutant	Symbol	Concentrations (ppm)
Cyanide (total)	CN(T)	462
Cyanide (amenable to chlorination)	CN(A)	459
Sulfide	S	0.90
Fluoride	F	2.10
Arsenic	As	0.02
Barium	Ba	0.04
Cadmium	Cd	24.8
Chromium (total)	Cr	0.12
Copper	Cu	52.5
Nickel	Ni	11.9
Silver	Ag	0.01
Zinc	Zn	11.2
Methylene chloride		0.01
Chloroform		0.02
bis-(2-ethylhexyl)phthalate		0.02

retention times. Based upon the results of the bench-scale studies, pilot-scale treatment studies were conducted to determine treatability of the F007 waste.

Standard procedures described in the EPA Office of Solid Waste Manual (Ref. 3) were strictly followed in the analytical procedures. Metal analysis was performed on an Inductively Coupled Plasma (ICP) Spectrometer while organics were analyzed on a Gas Chromatograph/Mass Spectrometer (GC/MS).

A) Bench-Scale Testing:

After a complete characterization of the F007 waste was performed, five distinct treatment options were developed for the purpose of optimizing the treatment of this waste. These treatment strategies were simulated at bench scale. All sludges and solids were settled and filtered prior to ion exchange to avoid fouling and clogging of the resins. In the laboratory, mixed-media filtration was simulated by Whatman filter paper 934-AH. Alkaline chlorination and lime precipitation were performed in batch mode while ion exchange operations were performed in a continuous mode by using laboratory burettes. After decanting the clear liquid from the top, the residual sludge slurry, collected at the bottom of the settling flask, was filtered. The wet sludge cake was analyzed for cyanide and metals concentrations.

Initially, a conventional treatment train was developed. This treatment train included two-stage alkaline chlorination for cyanide destruction, lime precipitation for metals removal, mixed-media filtration, and cation exchange for metals polishing. Figure 2 shows a schematic of this initial treatment configuration.

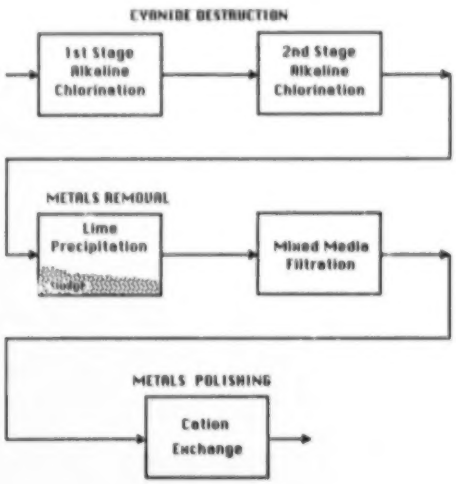

Figure 2. Treatment Train A - Conventional Treatment Train for Cyanide and Metal Bearing Waste

Results of this treatment train are presented in Table 2. Although this process configuration was effective in removing metals to an acceptable level, the cyanide concentration in the final effluent was higher than desired (18.7 ppm). The local sewer discharge limits for total cyanide is 10 ppm. In addition, the metal hydroxide sludge from lime precipitation contained approximately 2400 ppm total cyanide. Consequently, four more treatment trains (B, C, D, E) were developed to achieve lower cyanide concentration in the effluent and in the metal hydroxide sludge. These treatment trains included aspects of the following unit processes in different configurations: alkaline chlorination, lime precipitation, anion exchange for complexed cyanide removal, cationic exchange for metals removal, and precipitation of ferrocyanide complexes in the formation of Prussian Blue.

Table 2
Cyanide and Metals Analyses for Treatment Train A

Sample source	Concentrations (ppm)							
	CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag
Raw waste	365	29.3	0.04	93.7	14.6	0.01	13.1	0.04
Alk. chl.	17.0	29.6	0.05	66.0	14.5	0.02	12.7	0.04
Lime prec.	21.0	0.73	0.06	2.06	0.77	0.00	0.28	0.04
Cation exchange	18.7	0.01	0.07	0.02	0.23	0.01	0.00	0.02
Lime prec. sludge	2390							

A schematic of Treatment Train B is shown in Figure 3. This treatment train evaluates the effectiveness of cation exchange resin in removing

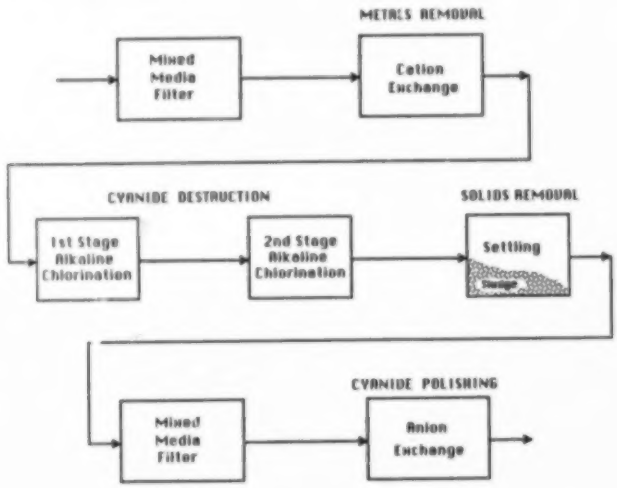

Figure 3. Treatment Train B - Metals Removal by Cation Exchange prior to Alkaline Chlorination

metals from the raw waste and segregating metals from sludges produced during alkaline chlorination. Alkaline chlorination and anion exchange follow the initial cation exchange metal removal step. Sludges produced during this step are settled and the supernatant filtered before anion exchange. Performance of this treatment train is shown in Table 3. Cation exchange in Treatment Train B failed to remove all the metals (Cu - 63.2 ppm; Ni - 11.1 ppm) and the majority of these unadsorbed metals precipitated in the alkaline chlorination sludge (as is illustrated in Table 3). In addition, the anion exchange resin failed to remove the cyanide in this treatment train (CN - 26 ppm).

Table 3
Cyanide and Metals Analyses for Treatment Train B

Sample source	Concentrations (ppm)								
	CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag	Fe
Raw waste	179	25.2	0.04	58.5	11.4	0.00	10.3	0.04	5.49
Cation exchange	288	0.58	0.01	63.2	11.1	0.00	0.17	0.05	2.99
Alk. chl.	22.0	0.89	0.02	61.1	11.3	0.00	0.22	0.06	4.92
Clarifier	29.0	0.28	0.02	1.36	1.45	0.00	0.03	0.04	6.48
Anion exchange	26.0								

Treatment Train C converts free cyanide into a ferrocyanide complex ($Fe(CN)_6^{4-}$) for subsequent removal by an anion exchange resin (IRA-958). Then, lime precipitation and cation exchange are employed for metal removal and polishing, respectively. A schematic of this treatment train is shown in Figure 4.

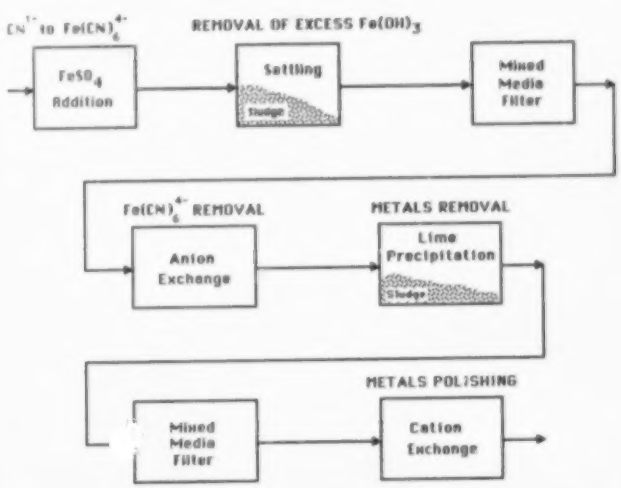

Figure 4. Treatment Train C - Initial Removal of all Cyanide in the form of a Ferrocyanide Complex using Anion Exchange

The objective of this treatment strategy was to completely remove the cyanide prior to lime precipitation with the hope that cyanide would be prevented from accumulating in the hydroxide sludge. Conversion of free cyanide into the ferrocyanide complex was achieved by dosing with ferrous sulfate ($FeSO_4$) at a pH of ~8.5. Excess ferrous sulfate forms ferric hydroxide precipitate ($Fe(OH)_3$) which was settled and filtered prior to anion exchange. Results of this treatment train are presented in Table 4.

Table 4
Cyanide and Metals Analyses for Treatment Train C

Sample source	Concentrations (ppm)								
	CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag	Fe
Raw waste	350	29.5	0.04	68.7	14.8	0.01	12.2	0.07	13.3
Anion exchange	25.0	1.41	0.00	0.30	0.00	0.00	0.05	0.04	0.50
Lime prec.	12.0	1.49	0.01	0.32	0.07	0.01	0.05	0.06	0.65
Fe(OH) sludge	9310	48.1	38.7	8.70	68.7	6.90	1800	4.40	38500

In Treatment Train C, again the anion exchange resin failed to remove all the cyanide (25 ppm CN in the effluent), part of which then precipitated with the hydroxide sludge. The $Fe(OH)_3$ sludge contained 9310 ppm CN, 48.1 ppm Cd, 38.7 ppm Cr, 68.7 ppm Ni and 1800 ppm Zn. With the accumulation of metals and cyanide in the hydroxide sludge this treatment option proved to be deficient in achieving desirable residuals separation of cyanide and metals.

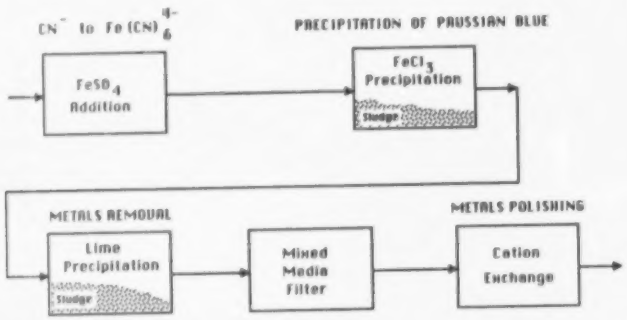

Figure 5. Treatment Train D - Initial Removal of Cyanide in the form of Prussian Blue

Figure 5 shows a schematic of Treatment Train D. The purpose of this option was to initially convert all free cyanide into the ferrocyanide complex, with subsequent precipitation in the form of Prussian Blue,

($Fe_4[Fe(CN)_6]_3$). Ferrous sulfate and ferric chloride ($FeCl_3$) are used in the two reactions at pH values of 8.5 and 6.5, respectively. Lime precipitation and cation exchange follow as metal removal processes. Table 5 exhibits the results generated from running this option. In Treatment Train D, the Prussian Blue precipitation step failed to remove

Table 5
Cyanide and Metals Analyses for Treatment Train D

Sample source	Concentrations (ppm)								
	CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag	Fe
Raw waste	350	29.5	0.04	68.7	14.8	0.01	12.2	0.07	13.3
Pr. Blue prec.	83.0	0.44	0.00	55.8	13.6	0.01	0.15	0.04	1.93
Lime prec.	83.0	0.70	0.04	58.7	11.6	0.05	0.17	0.09	1.49
Pr. Blue sludge	15600	1690	42.5	53.0	106	1.50	567	4.10	62600

all the cyanide (83 ppm CN in effluent). Furthermore, the sludge contained 1690 ppm Cd, 42.5 ppm Cr, 53 ppm Cu, 106 ppm Ni and 567 ppm Zn in addition to the 15,600 ppm CN and 62,600 ppm Fe.

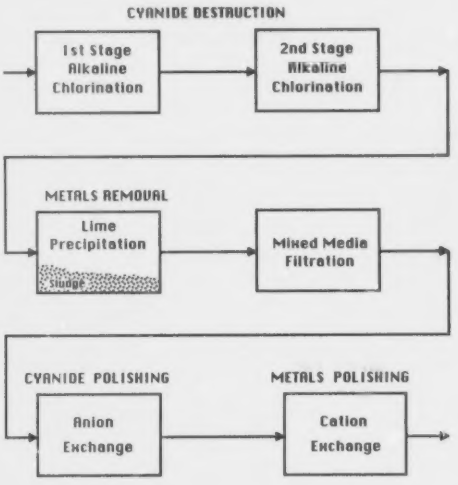

Figure 6. Treatment Train E - Cyanide Polishing with Anion Exchange Resins after Lime Precipitation

Treatment Train E, as shown in Figure 6, combines alkaline chlorination for removal of cyanide and lime precipitation for metal removal. Anion exchange is employed for the polishing of cyanide, (particularly the stable ferrocyanide complexes), while cation exchange

was used for metal polishing. As Table 6 shows, this treatment train achieved significant reduction of constituent metals and cyanide concentrations in the effluent.

Table 6
Cyanide and Metals Analyses for Treatment Train E

Sample source	Concentrations (ppm)								
	CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag	Fe
Raw waste	513	25.3	0.06	55.8	11.9	0.01	10.4	0.02	3.72
Alk. chl.	36.0	24.6	0.06	57.8	11.9	0.00	10.6	0.06	4.62
Lime prec.	30.0	0.48	0.05	1.08	0.32	0.03	0.24	0.05	8.69
Anion exchange	0.10	0.07	0.04	0.14	0.07	0.02	0.05	0.04	0.09
Cation exchange	0.15	0.05	0.05	1.04	0.07	0.01	0.05	0.02	0.03
Lime prec. sludge	2530	8410	3.70	22400	4270	4.00	3610	12.7	275

After analyzing the treatment data generated from the four treatment trains it was concluded that Treatment Train E provided the best treatment for this particular waste. Although unsuccessful in completely separating CN from the metals in the hydroxide sludge (2530 ppm CN), Treatment Train E exhibited the lowest CN concentration in the effluent (0.15 ppm). Metals concentrations in the cationic exchange effluent were below sewage discharge limits (POTW).

Triplicate testing of Treatment Train E was performed to demonstrate the reproducibility of the data. The average effluent quality with the standard deviations are presented in Table 7.

Table 7
Cyanide and Metals Analyses for the Triplicate Run of Treatment Train E

Sample source		Concentrations (ppm)								
		CN (T)	Cd	Cr	Cu	Ni	V	Zn	Ag	Fe
Final effluent (Cation exchange)	AVG.	2.55	0.12	0.05	0.83	0.34	0.01	0.07	0.02	0.11
	S.D.	3.15	0.11	0.01	0.28	0.19	0.01	0.04	0.00	0.06
Sludge (Lime prec.)	AVG.	2050	8250	5.30	21600	4040	4.30	3520	12.0	233
	S.D.	368	196	1.31	131	205	0.29	77.6	0.57	40.7

B) Pilot-scale testing:

Following the bench-scale evaluation of the various treatment trains, pilot-scale treatment of the F007 waste was conducted utilizing Treatment Train E. This discussion of the pilot plant performance involves system design, sampling procedure and final effluent quality. Figure 7 illustrates the pilot system schematic.

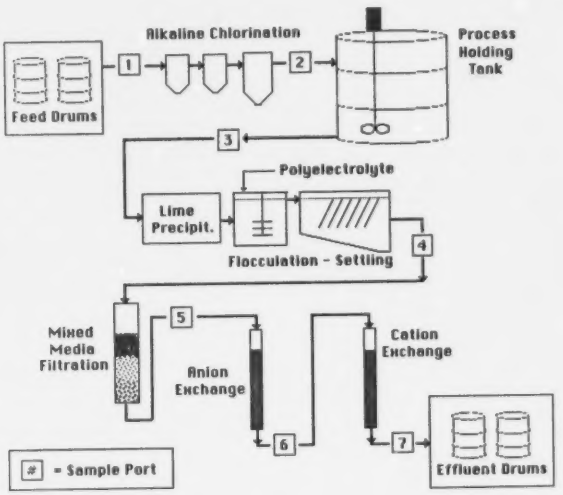

Figure 7. Schematic of the Pilot-Scale Treatment System

The pilot-scale system was designed for a flow rate of 1 gpm. However, due to waste quantity limitations, the flow rate in the alkaline chlorination units was reduced to 0.5 gpm. The complete treatment was performed in two days - alkaline chlorination on the first day and metals removal and polishing on the following day. All the effluent from the alkaline chlorination was collected in large Nalgene tanks integrated within the process train for overnight retention. Results from the bench-scale studies confirmed that increasing the retention time of the second stage from one hour to 24 hours does not have any significant effects on the cyanide concentration.

Sampling was performed allowing for unit process hydraulic retention times. For example, the effluent sample collected after the two-stage alkaline chlorination was taken two hours (the residence time for the two-stage alkaline chlorination process) after the corresponding influent sample. Similarly, the effluent sample from the mixed-media filter was collected 4.5 minutes (residence time for the filtration column) after the collection of the corresponding filter influent sample. At least seven paired data points were collected for each unit process. The sample port

locations are indicated in Figure 7. Samples were collected at a rate of one sample every 30 minutes at each sample port.

Testing was performed at the following optimum conditions, which were determined in prior optimization studies:

First stage alkaline chlorination:
- pH 11.5
- ORP 250 mV
- residence time 60 min.

Second stage alkaline chlorination:
- pH 8.5
- ORP 800 mV
- residence time 60 min.

Lime precipitation:
- pH 10.5
- residence time 15 min.

Flocculation:
- residence time 15 min.
- polyelectrolyte type cationic
- polyelectrolyte dose 20 mg/L

Clarification:
- residence time 30 min.
- loading rate 0.104 gpm/ft^2

Anion exchange for cyanide polishing:
- loading rate 1 gpm/ft^3
- resin type Amberlite IRA-958
- resin capacity 1.2-2.3 lbs CN/ft^3

Cation exchange for metals polishing:
- loading rate 1 gpm/ft^3
- resin type Amberlite IRC-718
- resin capacity 1 meq/ml. wet resin

The treatment train was effective in achieving very low concentrations of cyanide and constituent metals in the final effluent. The average cyanide concentration in the effluent was 0.91 ppm while the effluent concentration for each of the metals was < 0.4 ppm. The average pollutant concentrations in the effluent samples with standard deviations are given in Table 8.

Table 8
Cyanide and Metals Analyses for Pilot-Scale Testing

Sample source		Concentrations (ppm)							
		CN (T)	Cd	Cr	Cu	Ni	V	Zn	Fe
Raw waste	AVG.	285	27.3	0.03	55.5	12.7	0.01	11.2	3.99
	S.D.	67.5	0.50	0.00	1.64	0.23	0.00	0.17	0.69
Alk. chl.	AVG.	23.1	25.6	0.03	54.3	9.42	0.01	10.4	3.88
	S.D.	4.56	1.26	0.00	3.18	0.45	0.00	0.56	0.43
Lime prec.	AVG.	19.6	1.82	0.05	4.59	1.52	0.01	0.78	4.93
	S.D.	8.19	0.18	0.02	0.38	0.06	0.01	0.09	0.23
Anion exchange	AVG.	0.75	0.08	0.01	0.51	0.38	0.01	0.07	0.15
	S.D.	0.12	0.06	0.01	0.25	0.15	0.00	0.03	0.07
Cation exchange	AVG.	0.91	0.08	0.03	0.13	0.34	0.01	0.08	0.21
	S.D.	0.76	0.05	0.01	0.06	0.06	0.01	0.02	0.02

Table 9 presents the variation of the final effluent quality over time during the run. The variations are random and show no sign of any breakthrough in the ion exchange columns.

Table 9
Variations in the Final Effluent Quality over Time

Sample time (minutes)	Concentrations (ppm)							
	CN(T)	Cd	Cr	Cu	Ni	V	Zn	Fe
0	0.43	0.18	0.03	0.09	0.28	0.02	0.13	0.23
30	1.17	0.09	0.02	0.15	0.32	0.02	0.09	0.23
60	0.60	0.03	0.02	0.04	0.22	0.01	0.05	0.20
90	2.68	0.07	0.01	0.15	0.36	0.01	0.08	0.19
120	0.43	0.06	0.04	0.09	0.38	0.01	0.07	0.17
150	0.47	0.06	0.04	0.18	0.41	0.01	0.07	0.23
180	0.63	0.03	0.04	0.23	0.39	0.01	0.09	0.22
AVG.	0.91	0.08	0.03	0.13	0.34	0.01	0.08	0.21
S.D.	0.76	0.05	0.01	0.06	0.06	0.01	0.02	0.02

CONCLUSIONS

1. Two-stage alkaline chlorination coupled with anion exchange was able to polish a cyanide-bearing electroplating wastewater (F007) to the accepted discharge level for cyanide (<1 ppm of total cyanide).

2. Cation exchange polishing proved to be effective in removing residual metal species from the lime precipitation effluent (<0.4 ppm for each metal).

3. Examination of several process configurations utilizing alkaline chlorination and ion exchange determined that it was not possible to completely separate cyanide/cyanide complexes from metals in sludges produced in the treatment process.

REFERENCES:

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2. Code of Federal Regulations 40, Part 261, revised as of July 1, 1987.

3. USEPA, Office of Solid Waste, "Test Methods for Evaluating Solid Wastes" SW-846, 3rd edition, November 1986.